# HIGH RATE CONTINUOUS SYNTHESIS OF NANOCRYSTALLINE MATERIALS IN A COLLIDING VAPOR STREAM OF MICRODROPLETS

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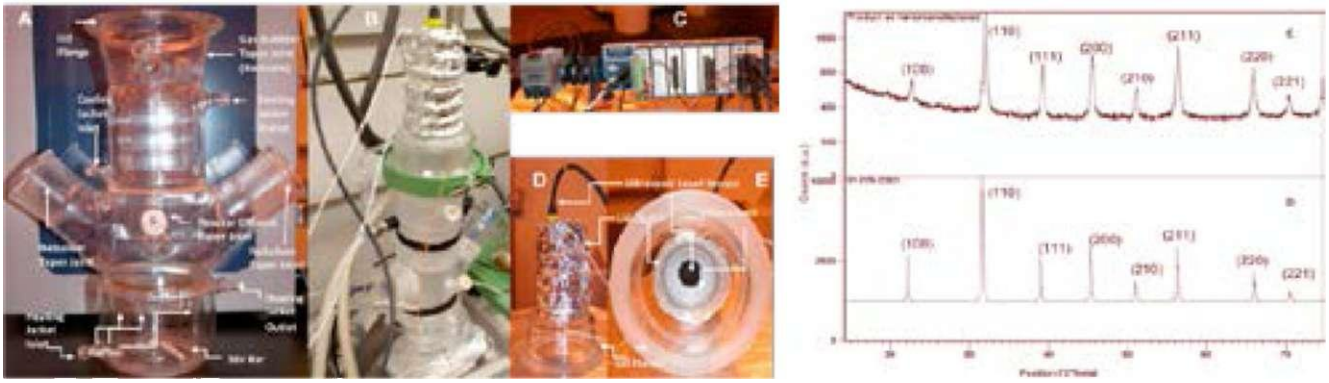
**Introduction.** Progress in nanotechnology is driving the need of large scale synthesis of functional nanomaterials. The lack of a workforce trained on process control and scale-up of nanomaterials manufacturing, the gap between laboratories and economically practical nanofabrication and the funding strain on the survivability of startup companies all contribute to the difficulties in scaling up nanotechnologies and their commercialization [1,2]. We report here a high rate continuous synthesis of functional inorganic nanomaterials using colliding vapor stream of reagents microdroplets.

**Materials and methods.** XRD pattern confirming the identity of cubic $BaTiO_3$ corresponding to JCPDS (00-079-2263) (D). Average particle size estimated to be ~23 nm from the Scherrer equation using the full width at half of the maximum intensity of the (110) diffraction peak.




***(A) Nanomanufacturing reactor composed of: two feeding ports.*** The main reactor chamber (0.350 L) is equipped with an internal reflux condenser, and six ports; 2 ports to hold the ultrasonic nebulizers to deliver a mist with tuneable droplet size. One port to hold the leveling sensor and two ports for inert gas supply and one port for product extraction. (B) Close-up photograph of the main reactor. C) Photograph of the data acquisition system (DAQ) that connects to a computer running LabVIEW via USB. D, E) The reactor lid showing the attached ultrasonic level sensor and electric heater. Side view of reactor lid (D); Bottom view (E).

**Results and discussion.** Our engineered reactor affords kilogram scale nanomanufacturing of sub-30 nm $BaTiO_3$ at neutral pH, atmospheric pressure, and at temperature below 100 °C. Furthermore, we were able to synthesize kg-scale $TiO_2$ oxides.

**Conclusions.** "Nebulization and collision of warm microdroplets (60-80 °C) of $Ba(OH)_2$ and $Ti(O\text{-}nBu)_4$ very quickly result in total hydrolysis and subsequent conversion to $BaTiO_3$, yielding 1.3 kg/day of high purity, highly crystalline nanoparticles (25-30 nm). This synthesis procedure also enables high-rate production of $TiO_2$ anatase (2.9 kg/day). It therefore provides a general platform for processing and scaling up of functional inorganic nanomaterials under very mild conditions" [2].

**Acknowledgments.** This work was supported by the US Army Research Office grant W911NF-09-D-0001 (ICB) and by UCSB's Center for Energy Efficient Materials, contract #DE-SC0001009. D.E.M. was supported by the US DoE (DEFG03-02ER46006). Materials were characterized using the facilities of UCSB's Materials Research Laboratory, NSF MRSEC Program grant no. DMR05-20414.

**References.**

1. T. Ould-Ely, M. Luger, L. Kaplan-Reinig, M. Doherty, D. Morse, NATURE PROTOCOLS, 2011, 6(1), 97
2. T. Ould-Ely, L-K Reing, D. E. Morse, 2014, ADVANCED FUNCTIONAL MATERIALS, 2014, 24, 1275